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Bis[4-(dimethylamino)pyridinium] hexachlorostannate(IV)

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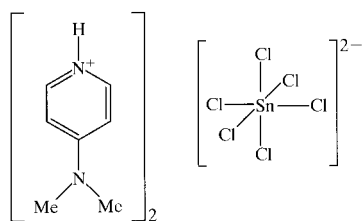
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In the title compound, 4-(dimethylamino)pyridine is protonated on the pyridine N atom. The N(CH₃)₂ moiety is twisted 4.4 (2)° from the pyridine-ring plane. The octahedral [SnCl₆]²⁻ anion is hydrogen bonded *via trans*-Cl atoms to pyridinium N atoms from two cations forming (C₇H₁₁N₂)₂[SnCl₆] structural units.

Comment

Synthesis of extended metal-halide systems could be achieved by the use of organic counter-cations as templating agents based on their shape and hydrogen-bonding capabilities. 4-(Dimethylamino)pyridine has two different N atoms susceptible to protonation, the pyridine and the amine. The pyridine N atom is the first choice for protonation in metal(II) halides (Halvorson *et al.*, 1990). The structural determination of the title compound, (I), was undertaken to examine cationation of



(I)

4-(dimethylamino)pyridine in the presence of metal(IV). The structure consists of [SnCl₆]²⁻ bridging two C₇H₁₁N₂⁺ cations *via* hydrogen bonding through two pyridinium N atoms. Protonation occurs only on the pyridine N atom. Atom N1 is π bonded to the pyridine ring with a short N1—C1 bond of 1.335 (4) Å, comparable to the ring's nitrogen contacts [N2—C4 1.335 (5) Å and N2—C3 1.322 (6) Å]. The dimethylamine moiety is basically planar with the pyridine ring, with only a 4.4 (2)° twist from the plane of the ring. The pyridine N2 atom is hydrogen bonded to Cl1 [N2...Cl1 3.231 (5) Å]. The pyridinium cations are oriented so as to lie in the *bc* plane and

form stacks parallel to the *a* axis.

Experimental

Colorless transparent needles of (I) were found as an impurity amongst the main yellow product 4-(dimethylamino)pyridinium tetrachlorocuprate(II) (Haddad & Willett, 2000). The source of tin was apparently a corroding spatula. The reaction medium contained 5 mmol of CuCl₂·2H₂O and 5 mmol of 4-(dimethylamino)pyridine in 20 ml 3 M HCl. The medium was heated at 348 K for 5 min. Yellow crystals of the copper(II) compound developed in two days together with few colourless crystals of the title compound of tin(IV).

Crystal data

(C₇H₁₁N₂)₂[SnCl₆]
*M*_r = 577.74
Monoclinic, *P*2₁/*c*
a = 8.3328 (17) Å
b = 11.680 (2) Å
c = 11.724 (2) Å
 β = 106.92 (3)°
V = 1091.7 (4) Å³
Z = 2

*D*_x = 1.758 Mg m⁻³
Mo *K*α radiation
Cell parameters from 4787 reflections
 θ = 1.98–28.19°
 μ = 1.911 mm⁻¹
T = 293 (2) K
Parallelepiped, colorless
0.40 × 0.20 × 0.15 mm

Data collection

Siemens SMART 1000 diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1997)
*T*_{min} = 0.528, *T*_{max} = 0.751
5296 measured reflections
1854 independent reflections
1629 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.035
 θ_{max} = 24.71°
h = -5 → 9
k = -13 → 12
l = -13 → 13
50 frames standard reflections
frequency: beginning and end of data collection
intensity decay: 2.4%

Refinement

Refinement on *F*²
R(*F*) = 0.025
wR(*F*²) = 0.065
S = 1.186
1854 reflections
162 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2 + 0.2141P]$
where $P = (F_o^2 + 2F_c^2)/3$
(Δ/σ)_{max} = 0.001
 $\Delta\rho_{\text{max}}$ = 0.32 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.34 e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.0036 (6)

All H atoms were located from a difference Fourier map and were refined isotropically.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); software used to prepare material for publication: SHELXL97.

The use of the single-crystal diffraction facility in the Office of Research at the University of Idaho is appreciated.

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